

## PATENT SPECIFICATION

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(72) Inventor JOHN WILLIAM GATISS

(54) IMPROVEMENTS IN AND RELATING TO FLUID FLOW  
CONTROL ARRANGEMENTS

(71) We, PYE LIMITED, of St. Andrew's Road, Cambridge, CB4 1DP, a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to arrangements aiding the control of fluid flow rates. In gas chromatography particularly, and in other techniques for small scale analysis or treatments, it is often necessary to provide and control streams of fluid with very low flow-rates. One way of doing this is to provide a pressure controller supplying fluid at a higher pressure and to place between this pressure controller and the device where the low flow is desired an impedance to fluid flow which induces a pressure drop related to the flow of fluid.

A known fluid restrictor element providing such an impedance is a mass of sintered porous material, for example sintered stainless steel, which can be positioned in the fluid supply. The pressure drop regulating the flow occurs at the restrictor element, which can be positioned some distance away from the pressure controller.

In gas chromatography, one situation where such a restrictor is useful is in reducing the pressure of a carrier gas into which is to be introduced a sample which will flow with the carrier gas stream into the chromatographic column. Another use is in reducing the pressure of the air and/or hydrogen which is introduced into a flame ionisation detector. Another use is in feeding the quench gas into an electron capture detector.

In such situations it would be advantageous to position the restrictor element near to the operative device to which gas is being supplied. One reason for this is that the restrictor could then be positioned in the

same temperature controlled zone as the operative device. Another reason is that if the restrictor is used to control ingress of gas into, for example, an electron capture detector, it is advantageous to have as little as possible dead volume of gas at low pressure between the E.C.D. and the restrictor, to prevent diffusion of sample substances backwards along the quench gas supply tube. It would also be advantageous for the member in which the restrictor element is positioned to be readily accessible for examination or replacement.

It is an object of the present invention to provide a fluid flow restrictor arrangement having one or more of the above advantages.

According to the invention there is provided a fluid flow restrictor arrangement for gas analysis equipment, including a tube through which gas may flow and a restrictor element to impede said flow secured in holding means which extends from one end of the tube, in which the holding means includes or consists entirely of a generally tubular member which is secured to the tube and which has a bore extending away from said end of the tube for communicating with an operative device, or another tube, to which the tube is to be coupled, and in which the restrictor element is a mass of porous material which has been packed by packing means against a shoulder at the end of said bore nearest the tube to secure the restrictor element in the bore, the degree of packing having ensured that in the absence of the packing means the restrictor element is sufficiently rigid to retain its shape and position in the bore and its required degree of impedance, the arrangement being such that disconnection and withdrawal of the tube from said operative device or other tube will cause the holding means and the restrictor element to be withdrawn with the tube so that the restrictor

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element can be removed from the equipment for examination or replacement.

According to the invention there is also provided a coupling arrangement for gas analysis equipment between an operative device of the equipment and a tube through which gas may flow, in which the tube is included in a fluid flow restrictor arrangement as defined in the previous paragraph; gas chromatographic apparatus including an operative device to which a tube is coupled by such a coupling arrangement; and a gas flow splitting device for gas analysis equipment including a chamber with at least one tube coupled to its respective entry or exit port by such a coupling arrangement.

The invention will be further described by way of example with reference to the accompanying drawings in which:—

Figure 1 shows a fluid flow restrictor arrangement including a tube and a restrictor element in holding means which extends from the end of the tube, according to the invention,

Figure 2 shows a variation, according to the invention, of the fluid flow restrictor arrangement shown in Figure 1,

Figure 3 shows a fluid flow restrictor arrangement which is closest in form to that shown in Figure 1 incorporated in a coupling arrangement according to the invention between the tube and a piece of apparatus to or from which fluid is passed,

Figure 4 shows schematically a gas chromatographic apparatus in which air and/or hydrogen is supplied to a flame ionisation detector via the coupling arrangement of Figure 3, and

Figure 5 shows schematically a gas flow splitting device for gas analysis equipment, according to the invention, with tubes coupled to respective entry and exit ports by coupling arrangements as shown in Figure 3.

Referring now to the drawings, in Figure 1 a metal tube 1 through which gas may flow has an enlarged generally tubular holder 2 brazed onto its end and extending therefrom. A constriction is formed within the holder by an internal flange one side of which forms a shoulder against which the tube end seats. The other side of the flange forms a shoulder 3 at the inner end of a bore 4 within which is situated a mass 5 of porous sintered material. In this case the mass 5 is sintered stainless steel but it could be sintered brass or aluminium, or a sintered plastics such as P.T.F.E., or some other porous material. The mass 5 is placed within the recess and is then packed down with a suitable tool in such a way that (a) the entire cross section of the recess is occupied; and (b) the degree of the impedance to fluid flow is as required. The degree of impedance can be fairly accurately con-

trolled over a wide range by careful packing. The shoulder formed by the flange 3 prevents the sintered material 5 from entering the tube itself. The material 5 itself is secured within the bore 4 i.e. it is sufficiently rigid when the packing tool has been removed to retain its shape and position in the bore 4. In the case of a stainless steel mass the material 5 can only be removed by drilling out the bore 4.

Figure 2 shows an other arrangement of a porous fluid flow restrictor with a tube end, and similar numerals refer to similar items. In Figure 2 a simple tube 2 is brazed to the outside of the tube 1. The tubes 1 and 2 together form the restrictor holder, and the shoulder 3 is formed in the recess by the end surface of the tube.

Figure 3 shows a restrictor arrangement which is close in form to that shown in Figure 1 incorporated in a coupling between the tube 1 and a piece of apparatus 6 to or from which fluid is passed. In a gas chromatograph such apparatus may be for example:— the air or hydrogen entry port of an F.I.D.; or the quench gas entry port of an E.C.D.; or one of the entry or exit ports of a gas flow splitting device; or the apparatus for injecting the sample into a carrier gas stream which passes along the tube 1.

The restrictor holder 2 is received in a recess 7 in the block 6, so that a bore in the block communicates with the bore of the tube 1.

The restrictor holder also acts as a securing and sealing member for the coupling between tube 1 and the block 6. As shown a hollow bolt 8 can screw into a threaded portion of the recess 7 to engage a shoulder 9 on the holder. The holder stands on an outer flange of the holder at right angles to the axis of the tube (and of the recess). A sealing washer 10 (of, e.g. Cu) may if required be placed as shown between the holder 2 and the end of the recess. The bolt 8 forces the holder against the sealing washer 10 or the end of the recess and thus securely couples the pipe to the block and causes a gas seal to be formed between them. The holder 2 could be exactly of the form shown in Figure 1, and in this case a shorter bolt 8 which fitted closely over the tube 1 and engaged the shoulder formed between the rear end of the holder 2 and the tube 1 would be used.

Figure 4 shows a typical application of the coupling arrangement shown in Figure 3 to the hydrogen supply for a flame ionisation detector in a gas chromatographic apparatus. The F.I.D. 11 is contained in a temperature controlled zone 12, in this case a detector oven, of the apparatus. The hydrogen is supplied from a variable pressure controller (not shown) which may be

operating in the range of 2-30 p.s.i. and may be situated at some convenient location in the apparatus through a 1/8 inch diameter tube 13 to a conventional pipe coupling 14 and thence to the tube 1 which in this case is 1/16 inch diameter. The coupling arrangement including a restrictor element 5 between the tube 1 and the F.I.D. 11 at the hydrogen entry port 15 is as shown in Figure 5 and is located in the same temperature controlled zone 12 as the F.I.D. Typical overall dimensions of the coupling arrangement between the tube 1 and the F.I.D. 11 are indicated by the length and diameter of the recess 7 being 3/5 inch and 1/10 inch respectively, the overall dimensions of the conventional pipe coupling 14 being typically 1 inch  $\times$  2/5 inch. The reduced pressure of the hydrogen in the F.I.D. is typically substantially equal to atmospheric pressure and the flow rate may be adjusted between 20 and 90 ml/min.

Figure 4 also shows schematically an arrangement of the other major features of a gas chromatographic apparatus. The sample to be analysed is introduced into a carrier gas at the inlet device 16 of a coiled chromatographic separation column 17 in a column oven 18. The detector 11 responds to the components of the sample eluted with the carrier gas from the column 17, and an ionisation current meter 19 measures this response.

Referring back to Figure 3, the joint between the tube 1 and the F.I.D. in the arrangement as described with reference to Figure 6 is on the low pressure side of the restrictor element 5. Thus the seal is not under substantial gas pressure and the sealing washer 10 is, in this case, not needed.

It can be seen that the arrangement of the gas flow impedance device described can be conveniently incorporated in a pipe coupling. Moreover, such a device can be positioned anywhere that a pipe is or can be terminated, in particular it can be positioned directly at the inlet to any device to which the gas flow is to be supplied.

A sintered mass restrictor element of the type with which this invention is mainly concerned can usually be removed only by drilling out the recess in which it is held. It is therefore an advantage of this arrangement that the restrictor holder 2 can be readily removed for inspection by uncoupling the tube 1, and that the piece of apparatus (i.e. the tube 1) to which the restrictor element 5 is attached can itself be discarded or replaced if necessary.

Figure 5 schematically illustrates an example of a gas flow ratio splitter 21 which uses the last-mentioned advantage. Gas from a gas chromatograph column enters the splitter via a pipe 1a and a sintered mass restrictor 5a which may be arranged

in the coupling between pipe 1a and splitter 21 in the manner of Figure 3. The two exit pipes 1b, 1c from the splitter are similarly arranged, and lead gas to similar pieces of apparatus 22b, 22c respectively which are collectors, detectors or analysers for the eluent. The restrictor 5a may be mainly for filtering and cleaning purposes and may be arranged to cause only a slight pressure drop. The same type of pipework and couplings (e.g. as shown in Figure 3) may be used for each branch of the splitter 21. The degrees of impedance produced in each arm, and the relative mass flow rates along each pipe 1b, 1c will therefore depend on the size of the respective restrictor elements 5b, 5c and on the density of packing of each. These parameters can be varied widely within couplings of the same type.

The gas emerging from a gas chromatograph separation column along the pipe 1a may be required to be split in certain ratios for various purposes. For example one branch 1b may lead to a collector for the eluent, whereas the other 1c may lead to a detector. In this case the ratio required to be obtained at the splitter may be as much as 25:1 or 100:1. As another example, the purpose of splitting the gas flow may be to pass the eluent to different detectors or analysers. In this case the ratio required may be much lower e.g. 5:1 or 1:1.

With the present invention, the splitter arrangement can be adapted to operate to these different requirements by removing one or both of the tubes 1b, 1c with their attached restrictor holders and restrictor elements, and replacing them by tubes whose restrictor elements produce the desired impedance to gas flow.

#### WHAT WE CLAIM IS:—

1. A fluid flow restrictor arrangement for gas analysis equipment, including a tube through which gas may flow and a restrictor element to impede said flow secured in holding means which extends from one end of the tube, in which the holding means includes or consists entirely of a generally tubular member which is secured to the tube and which has a bore extending away from said end of the tube for communicating with an operative device, or another tube, to which the tube is to be coupled, and in which the restrictor element is a mass of porous material which has been packed by packing means against a shoulder at the end of said bore nearest the tube to secure the restrictor element in the bore the degree of packing having ensured that in the absence of the packing means the restrictor element is sufficiently rigid to retain its shape and position in the bore and its required degree of impedance, the arrangement being such that disconnection and withdrawal of the

tube from said operative device or other tube will cause the holding means and the restrictor element to be withdrawn with the tube so that the restrictor element can be removed from the equipment for examination or replacement.

2. An arrangement as claimed in Claim 2, in which the generally tubular member fits around the outside of the tube where it is secured thereto.

3. An arrangement as claimed in Claim 2, in which the holding means consists entirely of said generally tubular member, in which said shoulder against which the restrictor element has been packed is formed by one side of an internal flange of said generally tubular member, and in which the end of the tube seats against a shoulder formed by the other side of the internal flange.

4. An arrangement as claimed in Claim 2 or Claim 3, and in which the generally tubular member is secured to the tube by being brazed thereto.

5. A coupling arrangement for gas analysis equipment between an operative device of the equipment and a tube through which gas may flow, in which said tube is included in a fluid flow restrictor arrangement as claimed in any one of Claims 2 to 4, in which said operative device of the equipment has a gas port including a recess shaped to receive said generally tubular member, and in which means are provided to detachably hold the generally tubular member in the recess so as to couple the tube to the operative device.

6. A coupling arrangement as claimed in Claim 5, and in which said means to hold the tubular member comprises the inner end of the recess which is shaped to seat one end of the tubular member and a detachable securing member adapted to bear against a surface on the generally tubular member which faces outward with respect to the recess.

7. A coupling arrangement as claimed in Claim 6, and in which the detachable securing member is a hollow bolt located on the tube and adapted to extend into the recess and engage an internal thread thereof.

8. Gas chromatographic apparatus including an operative device to which a tube is coupled by a coupling arrangement as claimed in any one of Claims 5 to 7.

9. Gas chromatographic apparatus as claimed in Claim 8, in which said operative device is a detector responsive to the components eluted with a carrier gas from a chromatographic separation column, and in which said tube is arranged to feed a gas required for the operation of the detector from a high pressure source of supply into the detector at a pressure reduced by the restrictor element.

10. Gas chromatographic apparatus as claimed in Claim 9, and in which a temperature controlled zone of the apparatus contains both the detector and said coupling arrangement whereby the restrictor element is subject to the same temperature control as the detector.

11. A gas flow splitting device for gas analysis equipment including a chamber with an entry port and two or more exit ports to each of which a tube is coupled, in which at least one said tube is coupled to its respective port by a coupling arrangement as claimed in any one of Claims 5 to 7 whereby the ratio obtained at the splitter can be changed by removing said tube with its respective restrictor element and replacing it by a tube with a restrictor element having a different impedance to gas flow.

12. A fluid flow restrictor arrangement substantially as described herein with reference to and as shown in Figure 1 or Figure 2 of the accompanying drawings.

13. A coupling arrangement for gas analysis equipment substantially as described herein with reference to and as shown in Figure 3 of the accompanying drawings.

14. Gas chromatographic apparatus substantially as described herein with reference to and as shown in Figure 4 of the accompanying drawings.

15. A gas flow splitting device for gas analysis equipment substantially as described herein with reference to and as shown in Figure 5 of the accompanying drawings.

C. A. CLARK,  
Chartered Patent Agent,

Century House,  
Shaftesbury Avenue,  
London, W.C.2.

Agent for the Applicants

Fig.1.

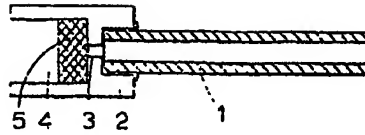


Fig.2.

